OIPE
OCT 16 2006
IAP85
PATENT & TRADEMARK OFFICE

TS6347US
SNO:SWT

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to Commissioner for Patents, Alexandria, VA 22313-1450, on or before the date shown below

Eugene R Montalvo
Date: 11 Oct 2006

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

| | |
|---|---|
| In re Application of: | |
| COENEN, Josef Guillaume Christoffel | |
| Serial No.: 10/519,137 | Group Art Unit: 2856 |
| Filed: 22 December 2004 | Examiner: Rodney T Frank |
| SYSTEM FOR DETECTING GAS IN A WELLBORE DURING DRILLING | 28 September 2006 |

COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450

## DECLARATION UNDER 37 CFR 1.131

Dear Sir:

I, Josef (John) Guillaume Christoffel Coenen hereby declare:

THAT I am the sole inventor and have signed the Declaration stating that I believe to be the original first and sole inventor in respect of the captioned present Application; and

THAT I was and still am employed by Shell International Exploration and Production BV which has company ties with the Assignee of the present Application; and

THAT I understand that certain claims in the present Application have been rejected as being anticipated by US Patent Number 6,675,914 to Masak, because the application underlying said US Pat. '914 was filed on 19 February 2002, which is before the priority date of the present Application; and

THAT I have conceived the invention underlying the present Application in The Netherlands, a WTO country; and

THAT I have conceived the invention underlying the present Application prior to 19 February 2002; and

THAT, as documentary evidence thereof, I attach hereto a document titled "Request for Patent Action" and excerpts from a document that was annexed to said "Request for Patent Action"; and

THAT I submitted the document "Request for Patent Action", together with said annexed document, to Shell Intellectual Property Department (IPS) in The Hague on 14 December 2001; and

THAT the document "Request for Patent Action" has been signed by me and my former Department Head, R. B. Stewart prior to its submission to IPS; and

THAT the attached pages show and describe detailed embodiments of the invention claimed in the present Application; and

THAT I have diligently worked with the in-house European Patent Attorneys of Shell Intellectual Property Services in the period between 19 February 2002 and 28 June 2002 to prepare and file European Patent Application 02 254596.6, from which the present Application claims priority benefits.

I further declare that all statements herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: October - 6 2006

Josef Guillaume Christoffel Coenen

CAU/ZEE/SOC/ CIR/ES/REP 1

BGS

CONFIDENTIAL

To be sent to: IPG/3 Formalities
The Hague

**REQUEST FOR PATENT ACTION**

To be completed by Intellectual Property Services

TS 6347

**OA Class(es):** 3A3
BUS CODE: EW
**Field Code:** WO
**Short Title:** Down-hole Electronic Nose sensor package for MWD.

**Date Received:** 14 DEC. 2001
**Attorney:** CAU

To be completed by Reporter

**Invention originating from:**
**Department:**
**Research Budget Code(s):** A-007858-100

**Brief Description of Research**

The reported invention entails a miniature electronic sensor package for literally "sniffing" hydrocarbon gasses down-hole in the re-circulation fluid stream from a drill. These gases are may be indicative for hydrocarbon charged prospects. The sensing package, hereinafter mentioned "Down-hole Electronic Nose", is able to measure methane ($CH_4$), carbondioxide ($CO_2$) and nitrogen ($N_2$), as free-gas or as gas dissolved in liquid. The Down-hole Electronic Nose is packaged such that forms an integral part of the drill and can be used for Measurement While Drilling (MWD) right behind the bit. The invention permits indication of hydrocarbon enriched subsurface layers while drilling obviating the need for tripping of the bit and to do subsequent logging. Business benefits are mentioned when using this technology specifically for E&P. The Electronic Nose sensor package is such miniature such that be placed inside a 2" OD Ultra Slimhole Drillstring (USD).

Further detailed decription of the invention is provided in document file:
<<Patent filing request E-Nose long.doc>>

**Name(s) of Contributor(s): Josef, Guillaume, Christoffel Coenen**
***Nationality* : The Netherlands**
***Business Address* :** Shell International Exploration and Production B.V.
Volmerlaan 8, Postbus 60, 2280 AB Rijswijk, The Netherlands
Tel: +31 (0)70 311 3108 Fax: 3110 Other Tel: +31 (0)79 3316873
Email: j.g.c.coenen@siep.shell.com
Internet: http://www.shell.com/eandp-en

***Home Address (only for US inventors)***

| | | |
|---|---|---|
| **Are there any relevant reports and/or experimental evidence available? (If YES, please attach copies)** | **EP 2001-5442** | **YES** |
| **Are there any relevant patents or other literature known? (If YES, please attach details)** | **See file** <<Patent filing request E-Nose long.doc>> | **YES** |

Signature of Reporter

Name of Reporter J.G.C. Coenen EPT-AWO
Extension No.: 3108
Date: 14-Dec-2001

Signature of Department Head

Name of Department Head
RB STEWART

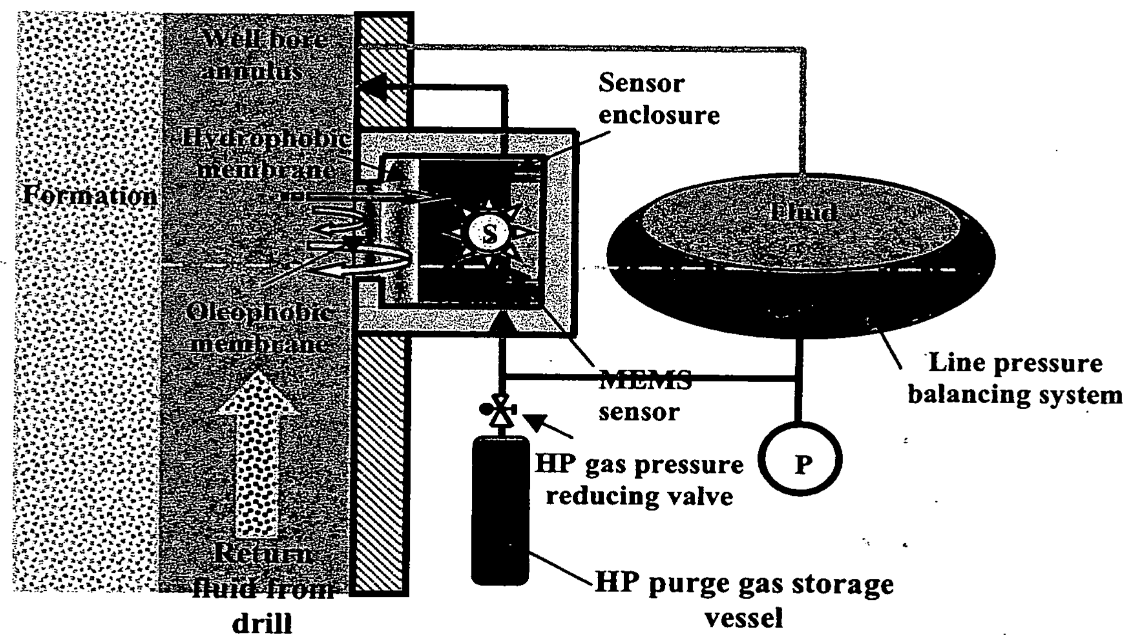
System description

The sensors of Down-hole Electronic Nose form the hart of the sensor package. The invention is based on utilising Micro-Electro-Mechanical-Systems (MEMS) solid-state sensors for this purpose. MEMS permit the mounting in cramped spaces and have very low power demands. The invention uses thermal conductive MEMS sensors. A drawback however is that MEMS gas sensors work only in a gas environment. Therefore the proposed method provides separation and extraction of gas from the re-circulation fluid of the drill in a down-hole environment still maintaining gas around the MEMS sensor element. Down-hole gas extraction should happen under high static pressure and temperature. The invention is based on a semi-open membrane connection between the sensor enclosure and the annular well bore space (between the drill string and the formation; see Figure 1). This membrane separation comprises a stack of a Hydrophobic (water repelling) and a Oleophobic (oil repelling), gas diffusion membranes mounted at the front of the sensor enclosure (see Figure1). The sensor enclosure volume is intermittently refreshed with Helium or Argon purge gas.
Gaseous components in the re-circulation flow from the drill passing the Down-hole Electronic Nose are have a phase identity of gas-in-solution or as free-gas bubbles. In either of the two cases the partial pressure for each individual gas component will be higher in the fluid than in the gas environment inside the sensor enclosure, which is almost pure Helium or Argon. Due to the difference in partial pressure between the external wellbore area and gas in the sensor room individual gas components will be driven by diffusion into the sensor room. This applies for every individual gas species in the gas diluted in the re-circulation fluid. Upon entering of gas inside the sensor room, the thermal conductivity of the gas environment around the MEMS sensor will alter and so does the sensor signal. After each measurement the sampled gas is removed by injecting Helium or Argon purge-gas from an on-board storage vessel. Therefore basically the MEMS based gas measurement of the invention is a differential measurement of a test gas relative to a reference purge gas. The sensor gas room of the Down-hole Electronic Nose assembly is small such (1) that only small volume of sample gas from the re-circulation fluid is needed in the sensor room for a quick measurement (2) the time for analysis is short and (3) that only a small volume of purge gas is needed to clean the sensor and to be ready for a next round.

Pressure balancing mechanism: By the capillary pressure inside the membranes a small pressure difference may exists outside versus the inside of the sensor housing. By choosing membranes with very small pores, this entry pressure may range from 2 - 14 bars. Nevertheless, at very large static pressure differences beyond 20 bar, such as is the case

down-hole, the water would invade the sensor enclosure and the sensors would be destroyed. Therefore for MWD application in E&P a need emerges for pressure compensation between the external (well bore pressure) and internal pressure such to be able to apply the system at large depth. The invention entails the mounting of a gas container with a flexible separation sleeve (see Figure 1). The space above the membrane is fluid filled and connected with the fluids in the well bore annulus. The part below the separation sleeve is gas filled and connected to the gas filled sensor room. By this arrangement pressure communication exists between the gas filled sensor enclosure and the fluid filled annular well. At the same time the flexible sleeve separates the well bore fluid from the gas in the sensor enclosure.
When the MWD sensor package lowers with the drill in the well, part of the purge gas, supplied from an on-board the high-pressure gas storage vessel, after reduction to the desired pressure level, will be consumed to counterbalance the increasing external pressure in the well bore. Thereto the high-pressure gas storage vessel for the purge gas should have a pressure beyond the maximal anticipated bottom hole pressure. In this way at all depths or all static pressure levels a pressure balance is kept over the vulnerable sensor membrane.

**Figure 1: Principle of down-hole electronic "gas sniffer" sensor package for gas-in-liquid detection at MWD.**




Sensor choice

During an exploration drilling operation the methane gas concentration may vary from very small levels i.e. 0% up to 100%. This requires MEMS sensors being able to accurately measure the gas concentration over the full range. Thermo catalytic MEMS pellistor sensors operate in a range from a few ppm up to 5 vol%. These sensors require oxygen or air to burn the gas sample which often will be methane. This can be done to 5 vol% below the lower explosive limit (LEL) for methane but above 5 vol% this will lead to explosion risks. Because in exploration drilling methane gas is the predominant gas species to be encountered, thermocatalyic sensors are considered to be potentially hazardous. On the other hand, thermal conductive MEMS pellistor sensors (MTCS sensors) can cope with the full range and are safe and are relative easy to operate as they just require to heat-up the gas sample a little. Therefore a thermal conductive pellistor sensor is proposed as an appropriate sensor for the Down-hole Electronic Nose package. A drawback however is that MTCS sensors are chemically not selective i.e. it just measures the thermal conductivity for the gas surrounding

the sensor. Standard MTCS sensors are often applied for fire protection and are tuned to the gas environment were it will be put in. The MTCS sensor is usually calibrated for binary gas mixtures.

The application of MTCS sensors involve purging of the sensor with a reference gas i.e. Helium or Argon. These gasses have extreme opposite thermal conductivity than any gas to be encountered during drilling. Upon diffusion of sample gas into the sensor room, the thermal conductivity of the gas mixture will change. As a result the output signal of the MTCS sensor will change and be indicative for the gas mixture around the sensor. The MTCS sensing principle in the invention basically is a differential measurement of the change in sensor signal due to a change in thermal conductivity of the gas environment.

Signal measurement strategy

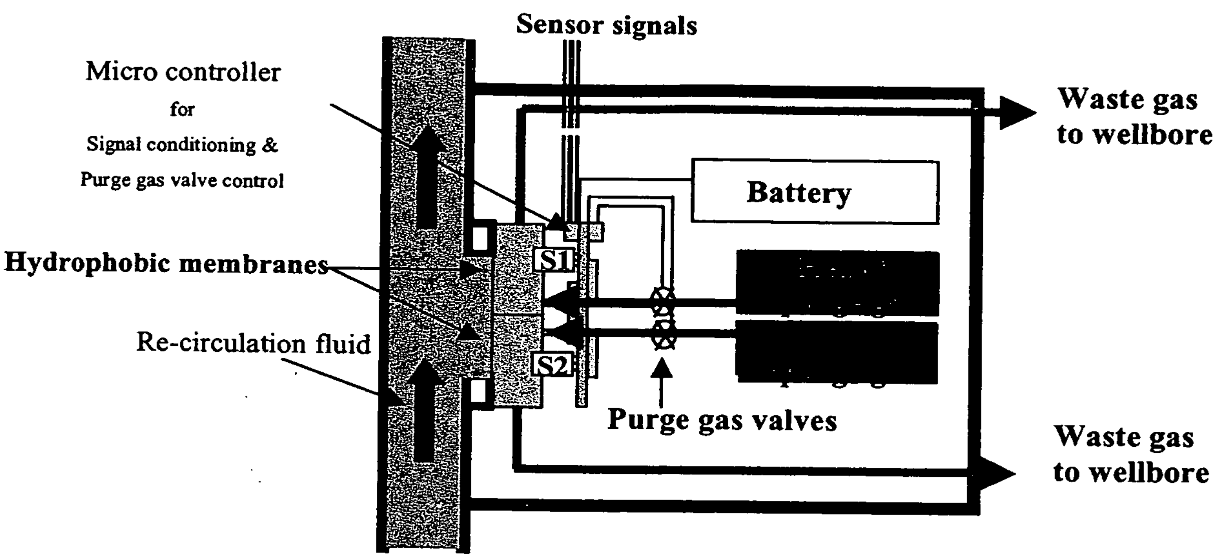
The measurement strategy with the Down-hole Electronic Nose can be two-fold. Method-1 involves utilization of amplitudes of output signals when, after an initial disturbance, the signal become stable. This requires a relative long time around 80 minutes for each measurement. Method-2 involves the measurement of the slope for the output voltage when it changes in time due to diffusion of sample gas into the sensor room. This is a fast measurement of around 15-20 s. For the Down-hole Electronic Nose we will follow method-2. However our claims will also incorporate method-1.

Method-2 is a fast measurement mode based on measurement of the slope of the sensor output signal. Each single measurement is carried out by alternating the purge gas and the measurement with 15 seconds interval. Cycle times are therefore 15/15 sec. The purge gas used in this case is Helium or Argon. Thereby large number of measurements that can be done in a short time frame i.e. 1 per 0.5 minute. In order to improve the accuracy statistical averaging can be carried out over large number of data samples. The slope is proportional to the partial dissolved gas concentration. Thereby the time needed to perform an analysis is reduced from 20 minutes to 15-30 seconds.
The slopes have been measured for 100% free gas as well as for gas fully dissolved in water and show significant difference to be used for recognition of the phase wherein the gas occurs. This can be either gas-in-liquid or as free gas in the mud return stream from the drill. Gas-in-liquid measurement, based on MTCS sensors in combination to semi-permeable membranes are possible.

Down-hole Electronic Nose configuration

Our invention comprises a sensor package comprising of two MTCS sensors (see Figure 2). The sensors are placed adjacently to each other in the same physical environment. Each sensor will have its own purge gas, one with Helium, the other with Argon. The sensor package is equipped with control valves and battery power module. The advantage would be that each sensor has its own purge gas and stable electronic settings (offset and gain).

**Figure 2: Dual purge-gas MEMS sensor package for down-hole gas sensing**




<u>Gas species discrimination method</u>

Purge gas is used to remove the waste gas after one measurement and to obtain a base line for a new measurement. Because the MTCS sensing principle is a differential measurement of the change in sensor signal due to a change in thermal conductivity of the gas environment. This principle can be used for the discrimination between $CH_4$, $CO_2$ and $N_2$.
Dual purge gas method: Basically the MTCS sensor arrangement offers one equation by measurement of the thermal conductivity of a mixture of three gas species at a certain fixed temperature of the sensor internals. A second equation follows by assuming that the sum of all concentration of 3 gas species $CO_2$, $N_2$ and $CH_4$ add up to one. In order to resolve 3 gas concentration a third equation is required. The invention provides a 3-th.equation for the gas mixture by carrying out a second identical MEMS thermal conductive measurement using a different purge gas. Thereto the Electronic Nose sensor package utilizes two purge gasses viz. Argon or Helium. Ecah sensor has its own electronic settings and gain such that signal levels are optimized for read-out and for solving the set of 3 equations and 3 unknowns.
(Possibly the 3-gas species detection problem can be reduced to a 1-gas species detection problem by making use of prior knowledge on the well. For instance by assuming that in a certain well the Methane concentration in natural gas is the only variable. However we are never absolutely sure that a signal variation may be due to a change in concentration of other hydrocarbon gas components than methane or from the influx of strange gas components).